Standard Operating Procedure

(SOP)

Metal-Supported Solid Oxide Electrolysis Cell Test

Test ID: HTE-P-13

Rev 1

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Approved By: NAME / TITLE Date

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Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
03/25/2019	All	First Release

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3. Procedures

a. Scope and Applicability – The purpose of this SOP is to describe the method for testing metal-supported solid oxide electrolysis cells. The test is carried out in a vertical furnace in a fume hood for safety related to the leakage of hydrogen.

b. Summary of Method – Test rig is cleaned and inspected before using. Pre-fabricated metal-supported solid oxide electrolysis cell is connected to a platinum mesh with conductive wires on the steam electrode side. Then the cell is sealed on the rig with glass paste (Schott GM31107). The air electrode is also connected to a platinum mesh with conductive wires. Hydrogen flow is passed through a water bubbler (glass jar) in a silicone oil bath to control the dew point temperature. The metal tube connecting the bubbler and the rig is wrapped with a heating belt.

c. Definitions – metal-supported solid oxide electrolysis cell (MS-SOEC); deionized (DI); hydrogen (H_2); platinum (Pt); open circuit voltage (OCV).

d. Health & Safety Warning – H_2 is flammable and explosive. Its flammability limits based on the volume of hydrogen in air at 1 atm are 4.0% and 75.0%. H_2 cylinder should be fastened away from the testing furnace (or any other heat source). Metal tubing (not plastic) should be used for H_2. Make sure there is no leakon the connectors. The testing furnace is located in a fume hood. Keep the doors of the fume hood closed.

e. Cautions –Flush the bubbler with H_2 to remove oxygen inside before connecting to the test rig. Flush the testing rig with nitrogen before connecting it to the H_2 tube. Install a pressure release valve (<10psi) for the bubbler to avoid shattering the glass jar in the event of over-pressure.

f. Interferences – The temperature of the bubbler affects the moisture concentration (steam content of the cathode feed). It needs to be calibrated with a thermal couple. The wrapping of metal tube (gas line carrying the steam) with a heating belt is important to prevent condensation. Make sure the heating belt temperature is higher than the temperature of the bubbler. Use four-probe test system to compensate for resistance in the conductive wires.

g. Personnel Qualifications / Responsibilities – Users have to be trained on the H_2 safety and wear safety glasses. Users also need basic knowledge of electrochemistry and understanding of electrochemical characterization methods prior to the test.

h. Equipment and Supplies – Bio-Logic potentiostat/EIS, Schott GM31107, ink vehicle (terpineol), DI water, home-made test rig

i. Step by Step Procedure

1. Check the test rig. Make sure it's not damaged during previous test. Polish the surface which will be in contact with the cell.
2. Prepare glass slurry: Weigh 12 g Schott GM31107 glass powder and 3 g ink vehicle and then mix them.
3. Connect the steam electrode to a Pt mesh with metal wire leads by spot welding.
4. Apply glass paste on the edge of the cell and the connecting part between the cell and the rig. Thermal treat it: 90 ◦C/0.2 h (10 ◦C/min), 200 ◦C/0 h (2 ◦C/min), 700 ◦C/1h (10 ◦C/min), cool down to room temperature (10 ◦C/min).
5. Connect the air electrode to a Pt mesh with metal wire leads by spot welding.
6. Apply the second layer of glass slurry and fire it with the same procedure.
7. Connect the conductive metal wires to the Bio-Logic potentiostat and monitor the OCV change. The OCV should be almost "0".
8. Flush the bubbler with H_2 to remove oxygen inside.
9. Flush the testing rig with nitrogen before connecting it to the H_2 tube. The OCV is increased to ~0.1 V.
10. Connect H_2 tube to the test rig. The OCV should be increased to ~1.1 V. Keep the H_2 flow to be 150 SCCM for 3% H_2O/H_2 and 75 SCCM for 50% H_2O/H_2. Keep an eye on the water level in the bubbler during test and add more DI water if the level is lower.
11. Set the temperature (81.7 ◦C for 50% H_2) of the silicone oil bath with a DI water bubbler inside. Wrap the metal tube with heating belt until the entrance of the furnace. Set the temperature of heating belt to be 30 ◦C higher than the oil bath.
12. Wait for equilibrium of the bubbler temperature and then do the test.
13. When the test is done and the furnace is cooled down to room temperature, de-assemble the cell from the rig. Using a diamond saw, cut off a small piece of glass seal, and use a sharp tweezer to peel off the whole cell.

- Instrument or Method Calibration and Standardization – Calibrate the temperature of the silicone oil bath and heating belt. Calibrate electrochemical work station, Thermocouple readouts, and flow meters periodically using manufacturer recommended procedure. Repeated experiments are required to confirm the quality of the data.

- Sample Collection – All the samples are solid after test. Cut off a small piece of glass sealing in fume hood and expose the edge of the cell. The cell can be peeled off by a sharp tweezer.

- Sample Handling and Preservation – No special handling is needed. Samples can be stored in air.

- Sample Preparation and Analysis – The cell is prepared by solution infiltration to load catalysts on both electrodes.

- Troubleshooting – If the OCV is much lower than 1.1 V with 3% H_2O/H_2, the cell or sealing could be leaking. If the OCV is higher than 0.97 V with 50% H_2O/H_2, the moisture concentration could be lower than 50%. This is because the bubbler temperature or the metal tube wrapped with heating belt temperature is lower than 81.7 ◦C.

- Data Acquisition, Calculations & Data Reduction Requirements – Extract Ohmic resistance and polarization resistance from EIS with EC-lab software. Calculate the degradation based on the initial and final voltage or current.

- Computer Hardware & Software – EC-lab software should be installed in computer, using with the Bio-Logic potentiostat hardware.

j. Data and Records Management – Cell and test parameters should be recorded on notebook and obtained data should be backed up.

4. Quality Control and Quality Assurance Section

Calibrate the oil bath temperature and heating belt temperature before applying the test. Monitoring the OCV changes. The final results should be in ±10% deviation.

5. Reference Section

Not applicable for this SOP.